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ABSTRACT

Described is an introductory materials science laboratory program which emphasizes crystal structure both on the atomistic and microscopic scale and the dependence of materials properties on structure. The content of this program is classified into four major areas: (1) materials science, (2) mechanical behavior of materials, (3) materials testing and evaluation, and (4) mechanical processing of materials. An introduction presenting the program objectives is also included. (HM)

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INTRODUCTORY MATERIALS LABORATORY

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AMHERST MASSACHUSETTS
JUNE 1971

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INTRODUCTORY MATERIALS LABORATORY

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Prepared for the American Society for Engineering Education Workshop
for Teachers of the First Course in Engineering Materials, June 23-25,
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I. Introduction

Since the coupling of theory and experimentation has always been the deciding factor in materials progress, the desirability of a strong laboratory program to complement the introductory materials courses cannot be over-emphasized. The objectives of the laboratory program are to:

1. Stimulate the student's intellectual curiosity by adding a sense of reality to the study of materials.
2. Start the development of the student's laboratory techniques and skills.
3. Develop the students' abilities to observe an experiment and to record the data in an accurate and systematic manner.
4. Develop the students' abilities to analyze experimental data.
5. Introduce the students to the ideas of process and theoretical models.
6. Develop the students abilities to write and present a technical report.

The possible content of the introductory materials laboratory can be roughly classified into four major areas:

1. Materials Science
2. Mechanical Behavior of Materials
3. Materials Testing and Evaluation
4. Mechanical Processing of Materials

A description of each of these type of laboratory programs is included in Part II of this paper. Which of these programs is "right" for a particular school would of course depend upon the nature and objectives of the lecture course. It is entirely possible that the "best" elements of several of these laboratory areas could be combined to optimally meet the needs of a particular school.

The key to any successful laboratory program is to get the students actively involved in the experiments. The normal means of teaching the laboratory is to have much of the equipment and materials assembled and ready to be used by the students in their allotted laboratory period. An instructor is generally available to explain the use of the equipment and the interpretations of the experimental results. The trouble that I have found with this conventional approach is that it stifles students' motivation toward independent and creative experimenting. Many of the students have a tendency to sit back and wait until the instructor and a few of the more eager students do the experiment for them. When something goes wrong with the experiment, the students perceive it as the instructor's fault and not as a problem that they have to solve and overcome. For the laboratory to be successful the students must be encouraged to use his own imagination in designing the experiments, experimenting, and interpreting the results.

The method that I have found to be most successful in teaching the materials laboratory is to devote the first 5-7 weeks of the term introducing the student to materials testing and examination and the laboratory facilities available. Also, one of the initial laboratory periods is devoted to the "Selection of Materials" in which the students are taught in part the use of the library. After these initial experiments are completed, the class is divided up into small groups of 2 or 3 students and each group then selects an experiment to do as a project. The projects are chosen from a list of experiments where the only information given is the project's objective, e.g. to determine the activation energy for grain growth of cartridge brass, and the pertinent references. It should be noted that the students are giving the option of working on a project of their own choosing if they so

desire and approximately 20 percent each term do so. At the end of the first two weeks of the project, the students have to hand in a project proposal that explains how they plan on accomplishing their objective. After receiving the proposal, the laboratory instructor generally has a 15 minute consultation with each group to go over any details that the students may have overlooked. At the end of the term the students have to hand in a formal project report and give a seminar (about 20 minutes) describing their project. The enthusiasm in which most of the students tackle their project has been most satisfying. Part of the success of this teaching method is undoubtedly due to having the students become actively involved in all stages of the project from the planning of the experiments to the analysis of results and the final write-up of the project.

Before concluding this section it is well worth noting that most laboratory programs are uninteresting, unimaginative, and uncreative. The major problems facing an instructor teaching the laboratory are:

1. Experiments must be available so that students can work in very small groups. This requires that inexpensive experiments and a large number of laboratory hours be used.
2. A few faculty members who are qualified and interested in teaching the laboratory must be made available and the system must be such as to utilize other faculty members or qualified graduate students where possible.
3. Communication between all faculty members and students must be done very efficiently or the faculty teaching loads will be excessive.
4. A critical amount of facilities, equipment, instrumentation, supplies, and technician's services must be available to the laboratory program. For example, the minimum facilities to teach a materials laboratory would be a universal mechanical testing system (preferably two or three), metallographic

facilities for examining the microstructure of materials, and furnaces (desirable to have one with an atmospheric control) for heat treating. To create a stimulating, challenging, and worthwhile laboratory program is a difficult task and requires a dedicated, enthusiastic, and imaginative faculty. I feel that the rewards are well worth the effort both in terms of being personally satisfying for the instructor and of greatly enhancing the engineering education of the student.

II. Laboratory Program

A. Materials Science

Event No. 224

American Society for Engineering Education
Annual Meeting, June 22-25, 1970
The Ohio State University
Columbus, Ohio 43210

INTRODUCTORY EXPERIMENTS IN MATERIALS SCIENCE

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Abstract

This paper describes an introductory materials science laboratory program which emphasizes crystal structure both on the atomistic and microscopic scale and the dependence of materials properties on structure. The experiments are divided into five groups: 1. Crystallography, 2. Crystals and Crystal Growing, 3. Solidification, 4. Cold Work-Annealing, and 5. Mechanical Properties - Multiphase Microstructures. Since the key to any successful laboratory program is to get the students actively involved in the experiments, the method that this author has found most successful is described.

INTRODUCTION

The teaching of materials has become primarily concerned with the fundamental principles which govern their behavior. The "Materials Science" course invariably emphasizes the relationship between the structure of a material and its behavior. The advent of such a course makes it necessary to devise suitable laboratory work in understanding crystal structure and how the structure of a crystal relates to its behavior. The experiments described in this paper are thought to accomplish these objectives. In general, the experiments are simple and do not require elaborate and complex equipment, with the exception of an x-ray diffraction machine.

CRYSTALLOGRAPHY

Crystallography is the science of the structure of crystals and knowledge of this subject is essential for a student studying materials science. Crystallography includes the ways of describing the structure of a crystal, the general principles that govern the types of structure that are possible, the structures of the crystals of specific substances, and the methods by which structures are determined. Crystallography is essentially a three-dimensional subject, and it is inherently difficult to represent three-dimensions on the pages of a book or blackboard. Therefore, it is most desirable for the students to handle, and better, to build three-dimensional models of crystals. The program learning series by Chalmers, et.al. [1], is excellently suited for these purposes.

In the laboratory, the students spend the first few weeks reading Books

1, 1A, 2, 3, and 6 of the program learning series. After each book is read, the student is given a few problems to solve which serve as a review of what he has learned. The students have received this series enthusiastically and their understanding of the corresponding material that is covered in the lectures has been enhanced. To date, the one major problem encountered with the program learning series is that the students, after reading Books 1 and 1A, can still not readily synthesize what they have learned in rationalizing observed crystal structures. To remedy this, a concise discussion of the rules governing crystal structures was prepared and a brief lecture illustrating these rules is given to the students after they had finished reading Books 1 and 1A. In addition, it was found that the well known "ball and stick" models of crystal structures could not be handled too extensively by the students for fear of breaking them. Also, there are difficulties inherent in stacking and cementing such models together. Because of this, large models were built with polystyrene foam balls and lucite sheet boxes. The lucite boxes were sized to produce correct stacking of close-packed structures when polystyrene balls of correct size are dropped into the box. Figure 1 shows a few of these models and characteristic inside dimensions are given in Table I. The ionic compound structures in Table I represent all of the close-packed AX structures given in reference [2]. Since the ionic crystal models were not completely self stacking, as were the BCC, FCC, and HCP models, it was found helpful to attach some of the polystyrene balls together with wooden pegs to add rigidity to these ionic structures. It should be noted that the CaF_2 structure (see Figure 1) was built so that the cation arrangement was FCC with the anions in all of the tetrahedral interstices. This was done so that the students could better understand how this structure can be visualized either in this manner

or as one in which the anions are arranged in a simple cubic arrangement with the cations in one-half of the 8-fold interstitial sites [2]. These models have become an essential part of the program learning series since the students can now handle the structures for better observation without danger of breakage.

CRYSTALS AND CRYSTAL GROWING

In order to bring the subject of crystallography closer to reality, the students grow various crystals such as NaCl, Rochelle salt, nickel sulfate hexahydrate, and sodium nitrate in the laboratory. An excellent description of how these crystals can be grown is given in reference [3]. The students not only find that the growing of these crystals is aesthetically satisfying but invariably become interested in the symmetry of the crystals and how internal symmetry can relate to external symmetry.

After growing the crystals, the students use the nickel sulfate hexahydrate and sodium nitrate crystals to demonstrate cleavage and glide [3]. The Rochelle salt crystal is used to demonstrate the piezoelectric effect by using the crystal to light a small light bulb [3]. The NaCl crystals are used in a powder diffraction experiment to determine the crystal structure and in a Laue back reflection experiment to determine the orientation of the single crystals grown.

SOLIDIFICATION

A study of solidification follows naturally from the experiments on crystal growth. Basis of this set of experiments are phenomena occurring in

low melting, transparent substances, particularly organic compounds. Being transparent, such substances can be observed by means of a microscope or micro-projector. Thus, these experiments can be either performed directly by each student or be used as part of a lecture-demonstration in the laboratory. Most of these experiments can be completed in a rather short time, 2 to 10 minutes. Reference [4] describes many of these types of experiments and some that this author has found useful are given below.

By fusing a few crystals of o-nitrophenol on a glass slide and covering, the solidification process can be viewed underneath a microscope. The crystals grow as many sharp tipped, lens-shaped needles that grow radially outward from centers of crystallization. A jagged pseudo grain boundary forms where two or more clusters of crystals meet. Micro-shrinkage may be observed between the individual needles. The liquid to solid volume change is evident in the drawing in of remaining melt as freezing proceeds. A shrinkage cavity or pipe forms at the end of the solidification interval and on further cooling the accompanying volume changes in the solid causes micro-cracking in the grain boundaries. By similarly observing the solidification of naphthalene colored lightly with methyl red, the rejection of the colored "impurity" by the growing crystals is seen. This simple experiment has formed the basis of a demonstration on the effects of zone refining by the author [5]. Dendritic growth of crystals can be observed on the freezing of quinone. Grains will develop within which one can see the ghosts of the dendritic mode of growth. Since the solidification of vanillin is quite sensitive to cooling rate, it represents an excellent material for demonstrating the effect that cooling rate can have on the final grain size. A moderate chilling, accomplished by placing a block of brass underneath the glass slide, will increase the nucleation rate and produce a finer grain size.

Grain growth after solidification can be studied using octachloropropane which freezes as polygonal grains which closely resemble brass or copper when examined in crossed polarized light. McCrone [6] discusses the grain growth of octachloropropane in considerable detail, including data on the rate of grain growth at various "annealing" temperatures, and suggests using this material for studying the mechanism of grain growth as influenced by strain and crystal orientation. This simple experiment serves as an introduction to the grain growth of brass experiment described in the next section.

This series of experiments is usually concluded with a phase diagram determination of the sodium chloride-water system. This diagram is published in Van Vlack [2] and can be determined from cooling curves of various composition sodium chloride solutions. The cooling curves are obtained by placing small beakers of solution in a coffee can of dry ice and measuring the cooling with an alcohol thermometer. The sodium chloride-water system is used along with the usual cadmium-bismuth and lead-tin systems because the students can observe that the onset of solidification does in fact correspond to thermal arrests.

COLD WORK-ANNEALING

The effects of cold work and annealing on the mechanical properties of copper and brass are well known and have become a standard laboratory experiment in many introductory materials courses. The cold worked and recrystallized materials can be examined microscopically to establish the inter-dependence of mechanical properties and microstructures. Results of the effect of annealing temperature and/or time on the mechanical properties of cold-worked copper and brass have been reported previously [7,8]. The

influence of cold work on the yield and tensile strength can also be studied and quantitative predictions compared to experimental results [9]. In addition, the grain growth of recrystallized brass can be investigated and compared to the theoretical grain growth law [10].

$$D^2 - D_0^2 = Kte^{-Q/RT}$$

where D = instantaneous average grain size

D_0 = as recrystallized grain size

K = constant

Q = activation energy for grain growth

R = gas constant

T = absolute temperature

t = annealing time (measured from start of annealing after recrystallization).

Experimental details are contained in reference [10] and student results are given in Figure 2. From these results the activation energy for grain growth of cartridge brass is calculated to be 25.6 Kcal/gram atom. For comparison, student results on the grain growth of octachloropropane are given in Figure 3 and the activation energy is calculated to be 1.74 Kcal/gram atom.

MECHANICAL PROPERTIES - MULTIPHASE MICROSTRUCTURES

To illustrate the dependence of mechanical properties on microstructure of multiphase materials and to introduce the student to one of the most important engineering materials, the carbon content of a range of steels can be related to yield stress, ultimate tensile strength, and percent elongation.

By examining each of the specimens microscopically, the student can establish the dependency of these mechanical properties on the distribution of the cementite phase. Student results on the effect of carbon content on the tensile properties of normalized plain carbon steel have been reported previously [7,8]. Also, the effect of cooling rate (or quenching mediums) on the microstructure of plain carbon steels and the resulting mechanical properties can be determined. Student results are given in Table II. References [8] and [11] give a good discussion of the inter-dependence of mechanical properties and microstructure of plain carbon steels.

Another excellent material for illustrating interactive properties of microstructures is Muntz metal (60% Cu and 40% w/o Zn). By solution treating Muntz metal samples at various temperatures in the α - β phase region and then quenching rapidly to preserve the equilibrium microstructure at that temperature, the amount of the β -phase may be varied. Since the percent β (which is the harder of the two phases) increases as the solution temperature increases, the hardness of Muntz metal increases with increasing solution temperatures. The hardness can be plotted as a function of both solution temperature and the percent β , which can be quantitatively estimated from the photomicrographs and compared to that predicted from the phase diagram. Muntz metal may also be age hardened. The aging treatment consists of heating samples to the all- β phase region and rapidly quenching to room temperature to retain the β -phase in metastable equilibrium. The samples are then reheated to various aging temperatures within the two-phase region, $\alpha + \beta$. At the lower aging temperatures the α -phase precipitates out in the form of very finely dispersed particles and a corresponding higher hardness is observed. At the higher aging temperatures, the α -particles coalesce and form globules of the α -phase during the aging treatment and hardness decreases. Reference [10]

gives an excellent discussion of the solution treating and age hardening of Muntz metal along with typical hardness results and photomicrographs.

METHODOLOGY

The key to any successful laboratory program is to get the students actively involved in the experiments. The normal means of teaching the laboratory is to have much of the equipment and materials assembled and ready to be used by the students in their allotted laboratory period. An instructor is generally available to explain the use of the equipment and the interpretation of the experimental results. The trouble that this author has found with this conventional approach is that many of the students have a tendency to sit back and wait until the instructor and a few of the more eager students do the experiment for them.

The method that has been found to be most successful in teaching the materials laboratory program described in this paper is to divide the class up into small groups of two or three after they have finished the program learning series. Each group then selects one of the remaining experiments to do as a project; however, the only information given to the students is the project's objective, e.g. to determine the activation energy for grain growth of cartridge brass, and the pertinent references. At the end of the first two weeks on the project, the students have to hand in a project proposal that explains how they plan on accomplishing their objective and the equipment and materials needed. At the end of the term the students have to hand in a formal project report and give a seminar (about 20 minutes) describing their projects. The enthusiasm in which most of the students tackle their project has been most satisfying. For example, this past term the two students

who selected the project of growing a Rochelle salt crystal to demonstrate the piezoelectric effect became so involved in the project that they built on their own time a functional loudspeaker with one of the crystals they grew [13]. Part of the success of this teaching method is undoubtedly due to having the students become actively involved in all stages of the project from the planning of the experiments to the analysis of results and the final write-up of the project.

SUMMARY

The group of introductory materials science experiments described in this paper emphasize crystal structure both on the atomistic and microscopic scale and the dependence of properties on structure. Although other series of experiments could be used, e.g. illustrating the mechanical behavior of the various classes of materials [7,8] or general metallurgy [14], the described set of experiments have been used this past year and have been well-received by the students. Part of the success of these experiments is believed to be due to the emphasis on correlating behavior with structure and the students being actively involved in the experiments.

ACKNOWLEDGEMENTS

This work was supported by National Science Foundation, grant number GY 4709. It is a pleasure to acknowledge the work of Mr. W. Dillahunty in organizing many of these experiments.

Table I.

Inside dimensions of lucite boxes for the indicated crystal structures.
 D is ball diameter; a is base length*; b is base width; c is height of container.

<u>Structure</u>	<u>Inside Dimensions</u>
HCP	$a = (1 + \frac{1}{\sqrt{3}}) D$ $c = (2\sqrt{6} + 1) D^{**}$
BCC	$a = b = (\frac{2\sqrt{3}}{3} + 1) D$ $c = 4 \text{ to } 6 a + D$
FCC	$a = b = (\sqrt{2} + 1) D$ $c = 4 \text{ to } 6 a + D$
CsCl	$a = b = \frac{(D_{Cs} + D_{Cl})}{\sqrt{3}} + D_{Cl}$ $c = 4 \text{ to } 6 a + D_{Cl}$
NaCl	$a = b = D_{Na} + 2D_{Cl}$ $c = 4 \text{ to } 6 a + D_{Cl}$
NiAs	$a = \frac{(D_{Ni} + D_{As})}{\sqrt{2}} + \frac{D_{As}}{\sqrt{3}}$ $c = 2\sqrt{3} (D_{Ni} + D_{As}) + D_{As}^{**}$
Zinc Blende (ZnS)	$a = b = \frac{2\sqrt{3}}{3} (D_{Zn} + D_S) + D_S$ $c = 4 \text{ to } 6 a + D_S$

Table I (continued)

<u>Structure</u>	<u>Inside Dimensions</u>
Wurtzite (ZnS)	$a = \sqrt{2/3} (D_{Zn} + D_S) + \frac{D_S}{\sqrt{3}}$ $c = 4D_{Zn} + 5D_S^{**}$
Corundum (Al_2O_3)	$a = \frac{(D_{Al} + D_O)}{\sqrt{2}} + \frac{D_{Al}}{\sqrt{3}}$ $c = 2\sqrt{3} (D_{Al} + D_O) + D_O^{**}$
$\gamma-Al_2O_3$	$a = b = D_{Al} + 2D_O$ $c = 4 \text{ to } 6 a + D_O$
CaF_2	$a = b = \frac{2\sqrt{3}}{3} (D_{Ca} + D_F) + D_{Ca}$ $c = 4 \text{ to } 6 a + D_{Ca}$

*For the structures based on a hexagon (HCP, NiAs, wurtzite and corundum) "a" refers to the side of the hexagon.

**This is 3 unit cells high.

Table II.

Effect of quenching medium on the mechanical properties of 1060 steel.

<u>Quenching Medium</u>	<u>Hardness</u>	<u>Ultimate Tensile Strength (psi)</u>
Furnace cool	76.5 R_B	71,000
Air	99 R_B	112,000
Oil	32 R_B	144,000
Water	62 R_B	320,000
Icy brine	62.5 R_B	325,000
Water (tempered at 600°F. for 1 hour)	49 R_B	248,000

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ADDENDUM June 23, 1971

A number of very excellent experiments concerning the electronic and magnetic behavior of materials are given in "Science of Materials Laboratory Manual," by G. Murphy, V.P. Denisen, N.C.J. Carlson, J.J. Keating, and J.H. McFadden of the Department of Nuclear Engineering, Iowa State University, Ames, Iowa, June 1967.

Some of the experiments described include Piezoelectric Effect, Ferroelectric Properties of Barium Titanate, Barrier Potentials, Work Function, Effect of Temperature and Impurities on the Conductivity of Ionic Crystals, Conductivity in Metals, Resistivity in Alloys, Reflectance of Metals, Properties of Semi-Conductor Materials, Point Contact Divdes, Hall Effect, Curie Point for Monel Metal, Ferromagnetic Domains, and Magnetostriction.

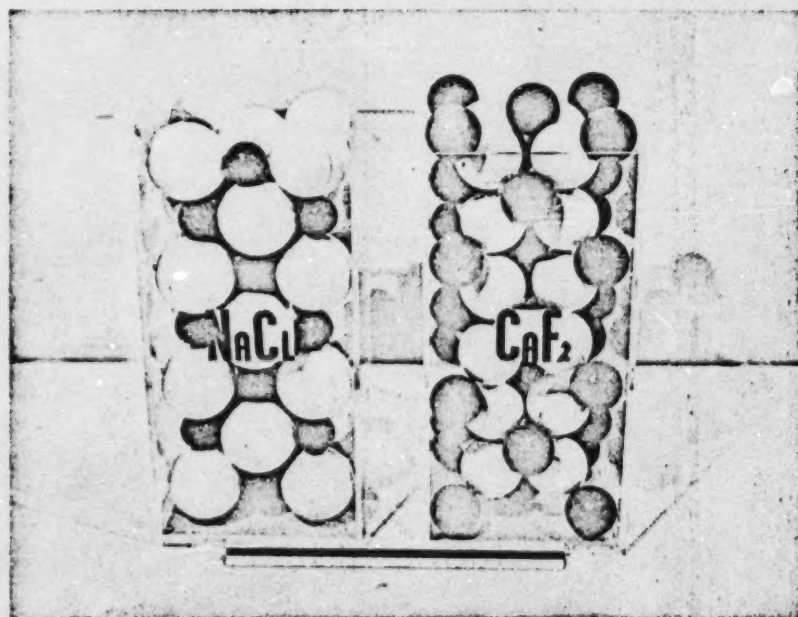
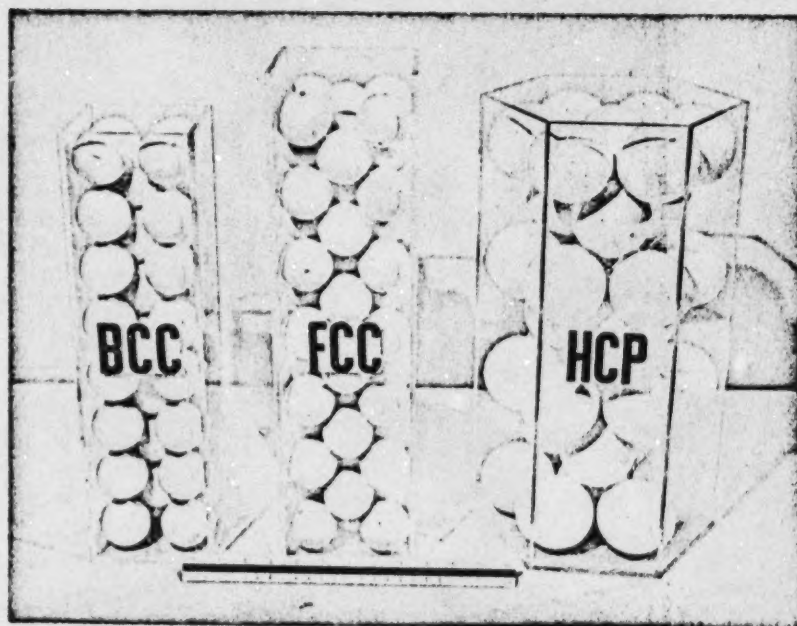


FIGURE 1. Crystal Structure Models.

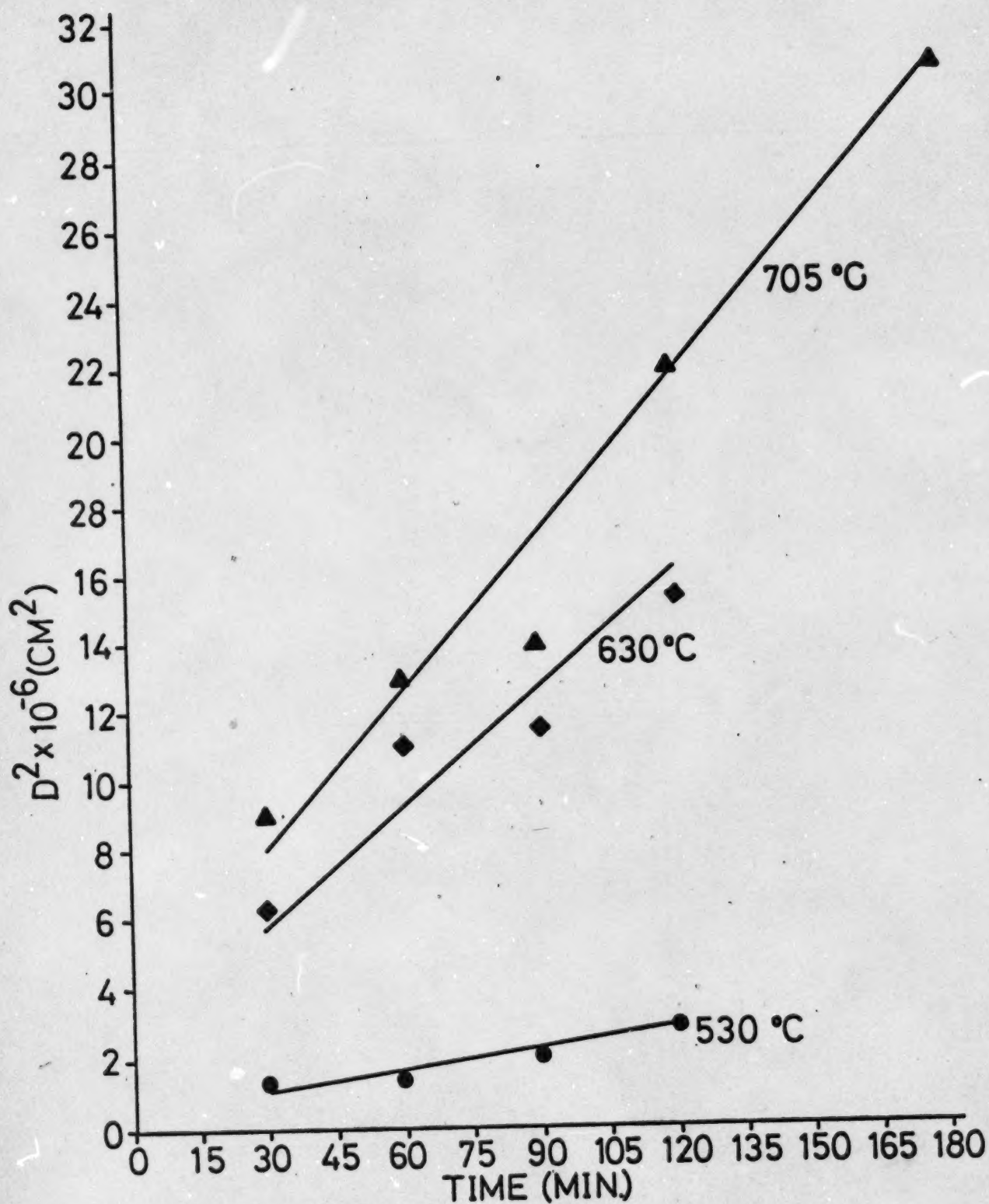


FIGURE 2. Grain Growth Results for Cartridge Brass.

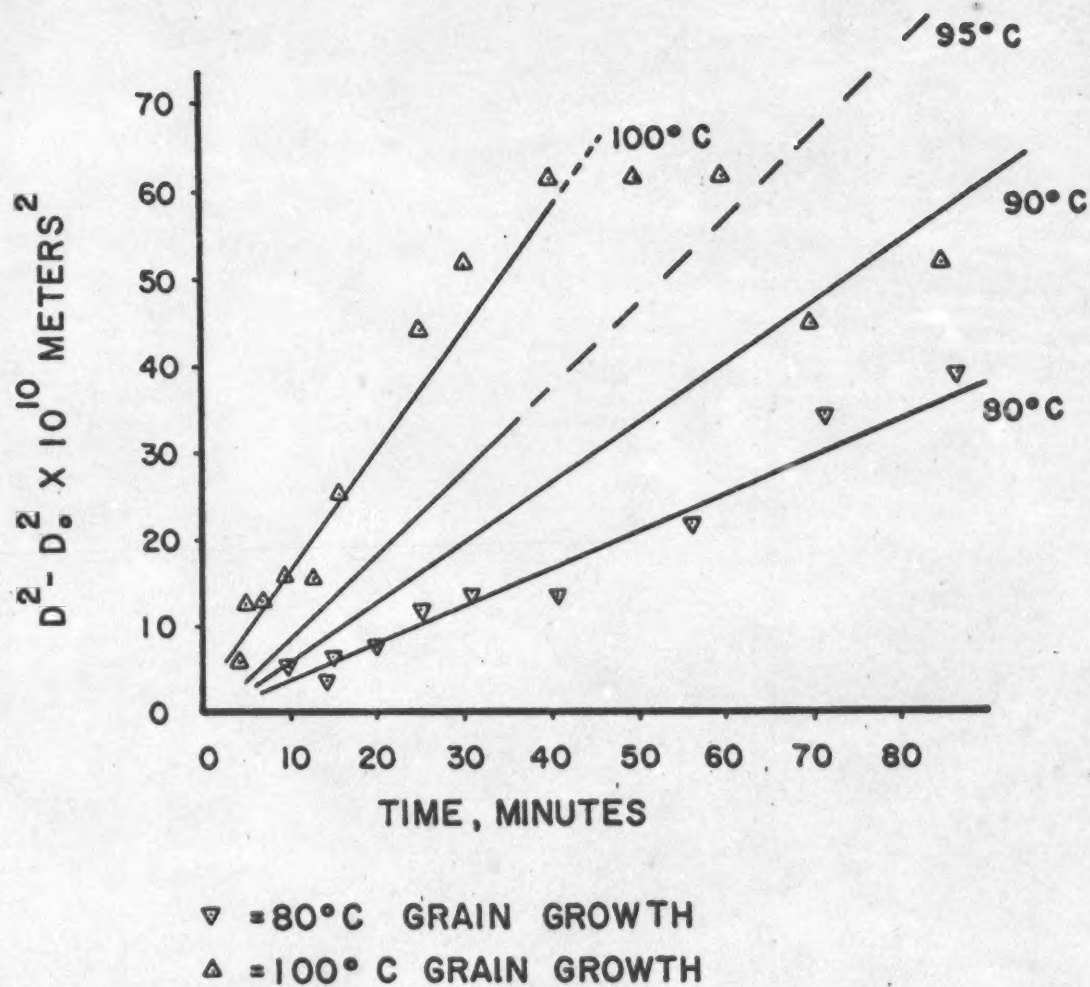


FIGURE 3. Grain Growth Results for Octachloropropane. Only the data points for the 80°C and 100°C are given for sake of clarity. The leveling off of the 100°C data after 40 minutes is thought to be due to the grains approaching their equilibrium shape and size.

B. Mechanical Behavior of Materials

INTRODUCTORY EXPERIMENTS IN MATERIALS SCIENCE FOR ENGINEERS

J. W. COAKHAM,* W. M. K. EASTWOOD* and W. EVANS*

(Received 22 July 1965)

INTRODUCTION

DURING the past few years, it has become increasingly obvious that the training of engineers, particularly at University level, should include a scientific study of engineering materials. To implement this, several Colleges have already introduced "Materials Science" into their curricula. The course invariably involves a consideration of the physical and chemical nature of materials, and an interpretation of material behaviour in these terms. The advent of such a course, where the main emphasis is placed on mechanical properties, makes it necessary to devise suitable laboratory work to supplement the lecture material.

With the exception of a tensile machine for testing rubbers, the experimental work presented in this paper has been devised using simple equipment which normally exists in most engineering departments, particularly if the department possesses a metallurgical laboratory. One of the major aims has been to utilize, as far as possible, the Hounsfield Tensometer, since its versatility, size and cost, fulfills the need for equipment which will allow large numbers of students to perform tests themselves.

Most of the material used has been purchased from stockists and converted into specimens by the simplest means possible. Inconsistency in material properties has been found, particularly between different batches, but it is considered that, with care, scatter of results can be reduced to a minimum.

This series of experiments is considered to represent a basis for student laboratory work in "Materials Science", and to illustrate the behaviour of materials, in such a way, that they fully endorse a lecture syllabus.

The materials used have been divided into three groups: (a) metallic materials, (b) high polymers, and (c) ceramic materials, and further sub-division has been made within these groups.

METALLIC MATERIALS

(1) A logical sequence for the presentation of the mechanical properties of metallic materials is to carry out a series of tensile tests to destruction on the following:

- (a) a pure soft metal,
- (b) a strain hardened pure metal,
- (c) a solid solution alloy based on the above metal, and in the annealed state,
- (d) a precipitation hardening alloy based on metal (a), in the annealed state,
- (e) alloy (d), heat treated to give maximum hardness.

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This experiment provides an illustration of improvements in strength brought about by increasing complexity of material, and also serves as an introduction to heat treatment. If the lecture syllabus were approached in this manner, giving a simple explanation of strain hardening in terms of the dislocation theory, then solid solution hardening and dispersion hardening would follow automatically.

Typical results using aluminium as the base metal are shown in Fig. 1. It can be seen that the semi-automatic recorder of the Tensometer is sufficiently sensitive to detect the serrated stress-strain curves associated with some alloys.

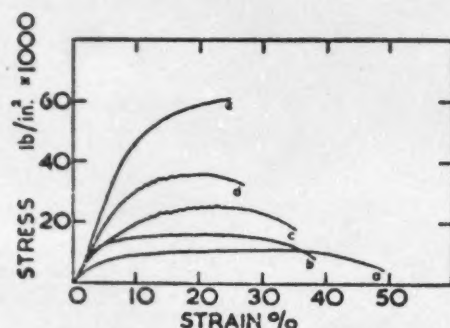


FIG. 1. Effect of various forms of hardening on the stress-strain characteristics of aluminium in tension. Rate of extension: 0.063 in./min.

- (a) Commercially pure aluminium in the annealed condition.
- (b) Commercially pure aluminium in the cold worked condition.
- (c) Aluminium-2 per cent magnesium (B.S. 1476 NE4) solid solution alloy in the annealed condition.
- (d) Aluminium-4 per cent copper (B.S. 1476 HE15) precipitation hardening alloy in the annealed condition.
- (e) Alloy (d), solution treated and aged for one week at room temperature.

(2) The effect of annealing on the mechanical properties of cold worked material has become almost standard in most metallurgical courses for engineering students. Hard drawn copper and 70/30 brass are readily available, while the small size of the specimens and their high conductivity makes it possible to remove specimens from a slowly heated furnace at appropriate temperatures. Only minor errors in the mechanical properties vs. temperature relationships will be introduced using this technique, and it has the advantage that the experiment can be completed in a single laboratory period of three hours duration.

The cold worked and recrystallized materials can be examined microscopically at this stage, to establish the inter-dependence of mechanical properties and microstructure.

This experiment has been used for the last two years, and the results shown in Fig. 2 are typical of those obtained by students in the metallurgical laboratory.

(3) Laboratory work in which the carbon content of a range of steels is related to yield stress, ultimate stress and percentage elongation, illustrates not only the important effect of yielding, but also introduces the student to the most important metallic material in engineering. The yield point phenomenon can be explained in lectures, and the dependence of strength on the pearlite constituent, as another example of dispersion hardening, can be further illustrated by microscopical analysis.

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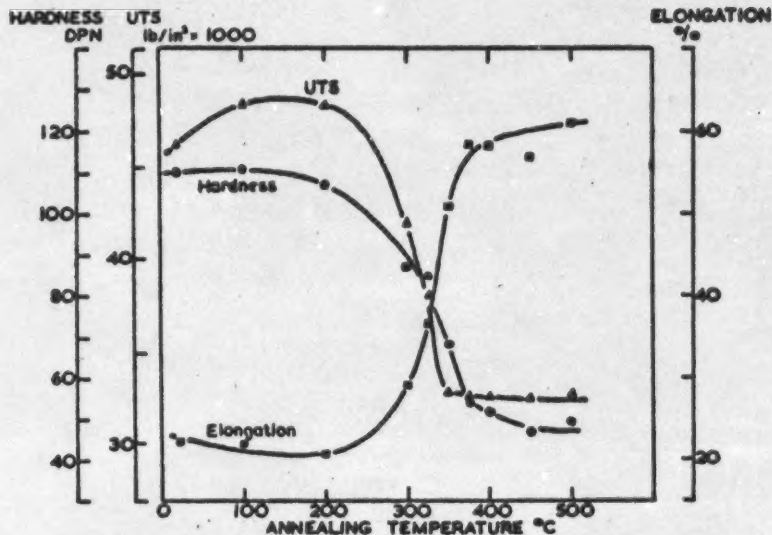
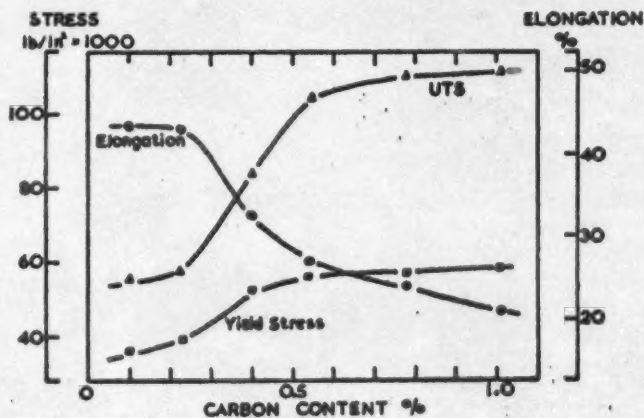


FIG. 2. Effect of annealing temperature on the mechanical properties of cold-worked copper.

FIG. 3. Effect of carbon content on the tensile properties of normalized plain carbon steels.
Rate of extension: 0.063 in./min.

Student results are shown in Fig. 3.

HIGH POLYMERS

(1) Rubber

With the considerable importance given to Hookean elasticity in engineering courses, this affords an excellent opportunity for studying non-linear elasticity.

Using approximately $\frac{1}{4}$ -in. square rubber, load vs. extension values can be recorded and converted readily to stress and strain by measuring changes in cross section. The non-

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linearity and hysteresis behaviour, coupled with the variation in Poisson's ratio with strain become apparent, as in Figs. 4 and 5 respectively.

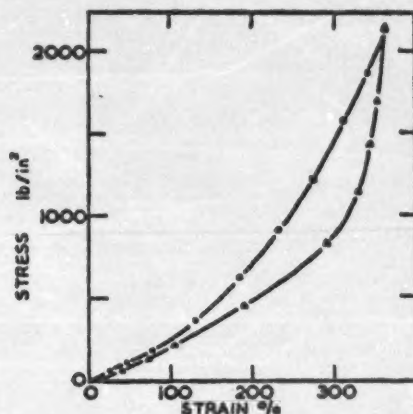


FIG. 4. Stress-strain relationship for natural rubber in tension.

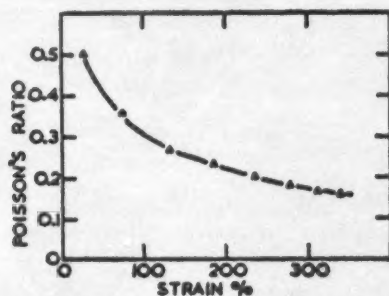


FIG. 5. Variation of Poisson's ratio with longitudinal strain during the progressive loading of rubber in tension.

(2) Thermo-plastic material

Specimens were cut from sheets using a high speed bench milling machine and a metal template. The tensile tests were carried out at an extension rate of 1 in./min, the specimens being held with the standard eccentric rubber chucks.

It had been intended to test destructively a range of materials of increasing molecular complexity, e.g. polythene, polyvinyl chloride, polystyrene, Nylon, Perspex, to show how the strength could be qualitatively related to molecular architecture, but because of the large number of variables in commercial grades of these products, no well-defined correlation of structure and properties could be established. In view of the uncertainty of comparing thermo-plastic materials on this basis, the experiments were confined to an investigation of the effects of: (i) density, and (ii) plasticizer, on the mechanical properties of thermo-plastics.

The effect of variation in density can be shown with high and low density polythene, typical results being given in Fig. 6. It was found that the high density variety exhibited

directional properties, so that specimens were always cut in the longitudinal direction. These directional properties themselves might provide an excellent basis for further experimental work on this and other materials.

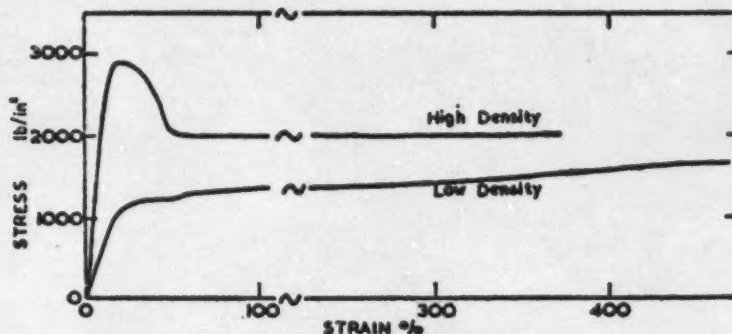


FIG. 6. Effect of density on the stress-strain behaviour of polythene in tension. Extension rate: 1.0 in./min.

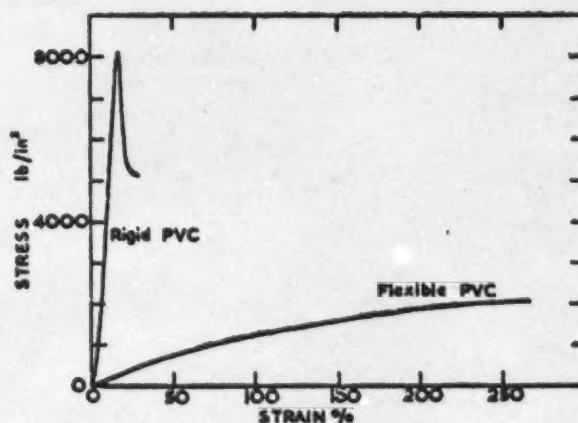


FIG. 7. Effect of plasticizer on the stress-strain behaviour of PVC in tension. Extension rate: 1.0 in./min.

Rigid (unplasticized) and flexible (plasticized) polyvinyl chloride were chosen to illustrate the effect of plasticizer. The partial neutralization of the intermolecular forces is a lecture topic which would be supported by the results shown in Fig. 7.

A more detailed study of the effect of plasticizer can be shown by investigating the variation in breaking strength and the elongation at fracture with percentage plasticizer, as indicated in Fig. 8. Also implicit in this experiment is the variation of modulus, which is analogous to the effect of temperature on unplasticized polyvinyl chloride. As results tend to be scattered, three specimens of each type were necessary, but such material with precise plasticizer content is difficult to obtain in commercial sheet form.

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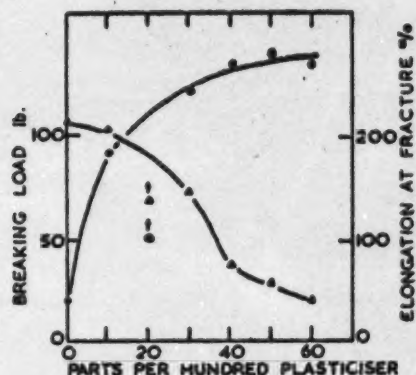


FIG. 8. Effect of plasticizer on the mechanical properties of PVC.

(3) *Thermo-setting materials*

Any metallurgist—and many engineers—will be familiar with the process of mounting metal specimens in Bakelite prior to examination. By filling and compacting the mould several times before curing, a suitable height to diameter ratio can be obtained in cured Bakelite samples for compression testing. If the degree of cross-linking is controlled by varying the temperature of curing, and the conditions during specimen manufacture are standardized, consistent results can be obtained. Figure 9 shows how the compressive strength varies with curing temperature. In this experiment, three specimens at each temperature were tested and the arithmetic mean taken, although the good reproducibility of results would permit students to use only one sample in each case.

The maximum temperature recorded during the manufacturing process, which was taken as the curing temperature, could not be maintained for any length of time; neither was this the true temperature of the Bakelite, since the thermometer was some half-inch from the actual sample. The temperatures are related however, and students could be encouraged to comment on experimental inaccuracies of this kind. Unfortunately, the size of the specimens made it necessary to use a testing machine whose capacity was much

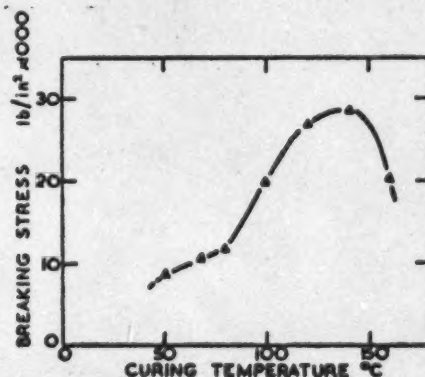


FIG. 9. Effect of curing temperature on the compressive strength of Bakelite.

larger than that of the Tensometer, but both the problem of size and temperature control could be remedied by the construction of a more refined piece of equipment.

CERAMIC MATERIALS

Without more elaborate equipment and extended laboratory time, investigations into the behaviour of ceramic materials become very difficult, particularly when trying to relate structure to mechanical properties. However, in order to complete the whole range of materials, two experiments may be introduced, although it is very doubtful whether the second bears any relation to the accepted theories of sintering.

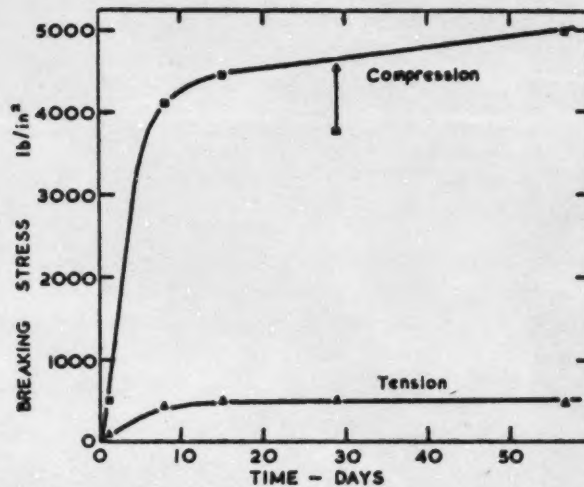


FIG. 10. Effect of ageing on the breaking strength of cement. (2 : 1 sand to cement ratio, 0.4 : 1 water to cement ratio).

(1) Civil engineering departments are already equipped to test materials such as cement and concrete, and some simple experiments on these materials can be included.

An investigation of the strength vs. time characteristics of cement has been carried out by students. The cement was mixed and compacted into standard briquettes, which were tested in tension, and also into 3-in. cubes for compression testing. These specimens were then tested after certain intervals of time, the results being shown in Fig. 10. The values in tension are the arithmetic mean of three tests, which show surprisingly good reproducibility, but unfortunately only one test was carried out in compression at each interval. The curves show, not only the ageing characteristics of cement, but also the vast difference between tensile and compressive breaking loads, which exists in ceramic materials in general.

(2) Laboratory work on the firing of ceramics is likely to be limited due to the high temperatures involved, but White Pottery Clay, having an optimum firing temperature of 1030–1100°C, is adequate for this purpose. The lower temperature can be achieved in a resistance wound furnace normally used for the heat treatment of steel. The clay was extruded into $\frac{1}{4}$ -in. diameter rod through a die with a long land, utilizing a Soil Mechanics screw type extruder, and then cut into specimens $\frac{1}{2}$ -in. high with a fine wire. They were

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dried for 24 h before transferring to the firing furnace, the temperature control of which was adjusted to reach the highest firing temperature of 1030°C in approximately $2\frac{1}{2}$ h. Specimens were removed at appropriate temperatures, and allowed to cool in air prior to testing in the Tensometer. Results for the experiment are shown in Fig. 11, the stress values again being the average of three readings.

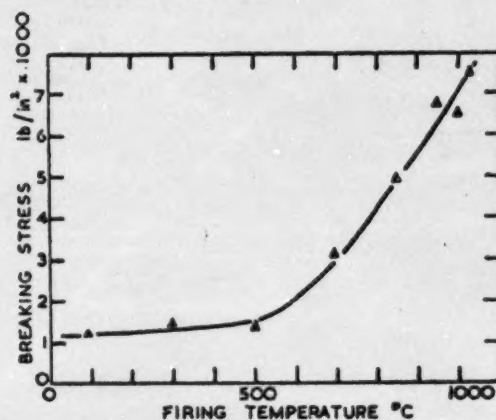


FIG. 11. Effect of firing temperature on the compression strength of White Potters' Clay. Compression rate: 1.0 in./min.

It is well appreciated that this method of firing does not allow reactions to be completed at any stage, but students can carry out the experiment in a three-hour laboratory period, and it is evident from the results shown that the essential principles of the firing process are illustrated.

STRENGTH-TEMPERATURE AND STRENGTH-EXTENSION RATE RELATIONSHIPS

(1) The effect of temperature on the strength of annealed copper is shown in Fig. 12, with individual stress-strain curves in Fig. 13.

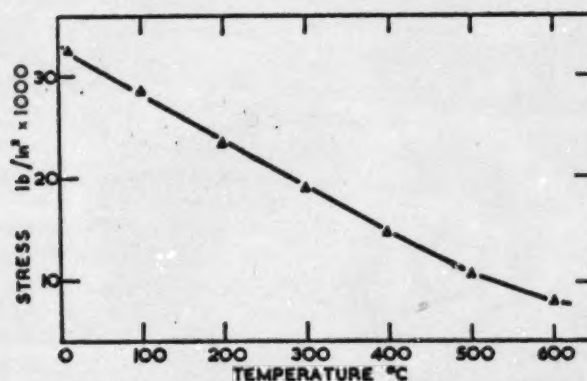


FIG. 12. Relationship between UTS and temperature for copper. Extension rate: 1.0 in./min.

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Furnaces and high-temperature chucks are available for use specifically with the Tensometer, but in order to prevent the specimens slipping under load, it was found that the chucks needed parallel lands for the collars. Also for a more accurate control of tempera-

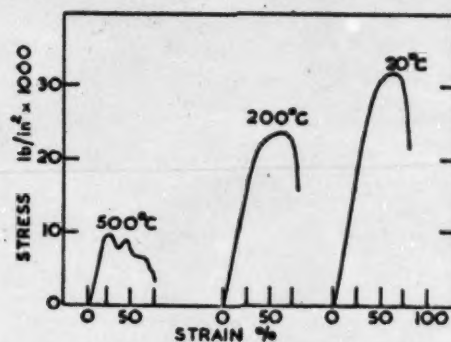


FIG. 13. Typical stress-strain curves for copper in tension at various temperatures. Extension rate: 1.0 in./min.

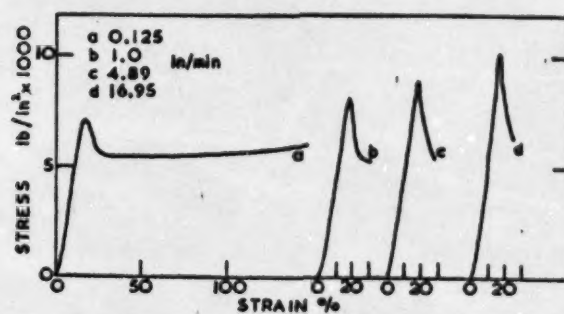


FIG. 14. Typical stress-strain curves for rigid PVC (Darvic) at various rates of extension.

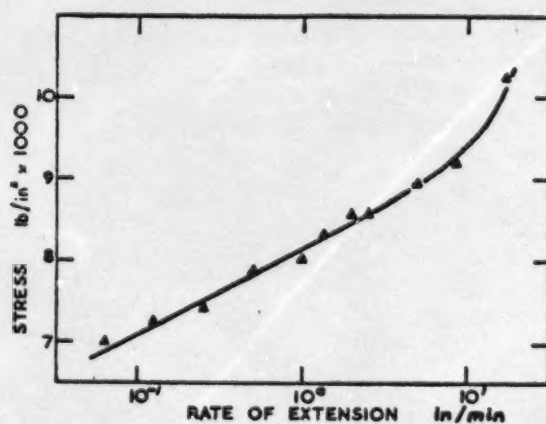


FIG. 15. Relationship between yield stress and extension rate for rigid PVC (Darvic) at room temperature.

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ture the Sunvic regulator was replaced by a variac. Temperature measurement was made with a mineral insulated thermo-couple placed in contact with the specimen.

It is probable that a similar relationship could be achieved with a thermo-plastic material, but for the small temperature ranges involved, a different heating system is required.

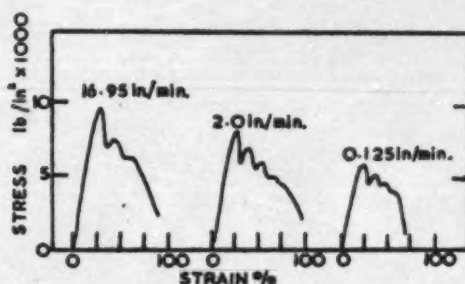


FIG. 16. Typical tensile curves at different extension rates for copper at 600°C.

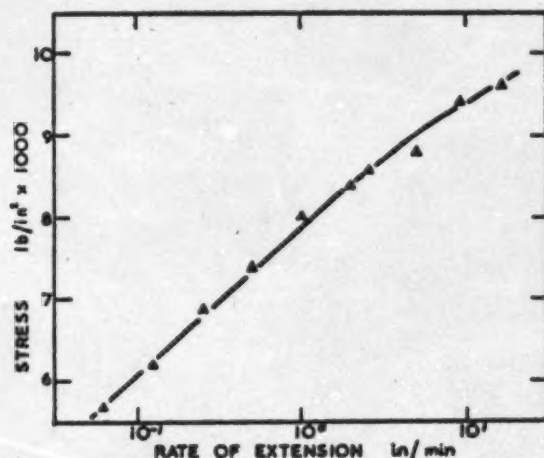


FIG. 17. Relationship between UTS and extension rate for copper at 600°C.

(2) The Tensometer can be adapted easily to give fairly high crosshead speeds by the use of suitable pulleys. Using these modifications, experiments were performed on rigid polyvinyl chloride at room temperature. Typical semi-automatically recorded stress-strain curves are shown in Fig. 14, while the variation of strength with extension rate for this material can be seen in Fig. 15.

Similar results for annealed copper at 600°C are given in Figs. 16 and 17 respectively.

In view of the unconventional curves in these latter experiments, particularly with copper at elevated temperature, it is suggested that the work be carried out merely to investigate the variation in some specific property or properties with temperature and extension rate, without attempting explanations of material behaviour.

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It is realized that this group of experiments is not comprehensive in that the emphasis has been placed on varying the material rather than the type of test. However, it does provide an introduction to materials behaviour, which can be elaborated according to the course of study.

Acknowledgements—The authors are indebted to Mr. F. C. SCOTT, Head of Civil and Mechanical Engineering Department, for the provision of laboratory facilities, and to Messrs. H. I. MILNTHORPE and ARMSTRONG for their constant encouragement.

ADDENDUM TO"Introductory Experiments in Materials Science for Engineers"

1. Metallic Materials

Additional ideas for experiments concerning the mechanical behavior of metals are included in the preceding laboratory program on Materials Science and in "Metal Science Book-Lab Kit," W.F. Collins and M.H. Richman, American Society for Metals (1963). Also, the stress corrosion of brass and mild steel can be studied. Ammonium hydroxide is quite effective in causing the stress corrosion of brass and ammonium nitrate for mild steel. It should be stressed that in all of these experiments on the mechanical behavior of metals the microstructure of the specimens should be examined to establish any property-structure relationships that may exist, e.g. yield strength dependence on say grain size or amount of ferrite.

2. High Polymers

It should be noted that Fig. 5 showing the variation of Poisson's ratio is incorrect since the authors used engineering strain for the definition of Poisson's ratio. If true strain had been used then Poisson's ratio would be a constant, 0.5, for all strains. For a discussion of this point please refer to "A Further Comment on Poisson's Ratio of Rubber," J.W. Coakham, et.al, Bull. Mech. Engng. Educ., 8, 191 (1969).

Two good inexpensive experiments dealing with the determination of the appropriate viscoelastic model for silicone putty and polyethylene are given in Mechanical Behavior of Materials, Frank A. McClintock and Ali S. Argon, editors, Addison-Wesley, p. 269 (1966). An excellent experiment on

the pressure-volume curve for a rubber ballon is described in "On the Inflating of Ballons," R.S. Stein, J. Chem. Educ., 35, 203 (1958). This is a perfect experiment to dramatically illustrate rubber elasticity.

3. Ceramic Materials

To illustrate the mechanical behavior of brittle materials, glass rods can be tested in three-or four-point bending. The dependence of fracture strength on surface condition (as-received, abraded, and acid-polished in a solution of 15% hydro-flouric and 15% sulfuric acid) can be measured and the size of the Griffith crack in each case calculated. Also, the dependence of strength on humidity can be demonstrated by testing the samples dry vs. wet and by measuring the strain rate sensitivity of the fracture strength on strain rate. Glass rods of soda-lime-silica and borosilicate (Pyrex) are readily available and illustrate the effect of glass composition on the strength. Additional experimental details are given in "Dynamic Fatigue of Soda-Lime-Silica Glass," J.E. Ritter, Jr., J. Appl. Phys., 40, 340 (1969). Since there is generally quite a bit of scatter in the fracture strength measurements of glass, these experiments offer an excellent opportunity to introduce the student to the statistical analysis of experimental data. Further, these experiments generally create quite a bit of interest and excitement. This is especially true for the acid-polished specimens where fracture strengths of 300,000 psi can be readily achieved and the glass very noticeably deflects before fracture and literally explodes when it fractures.

4. Dislocations

Many characteristics of dislocations can be observed utilizing two

pieces of plastic screen with a slightly rectangular grid. A grid of crossed screw dislocation, or edge dislocations, or combined edge and screw dislocation can be produced to give practice in analyzing the Burgers vector. Other experiments with the two pieces of plastic screening held in one's hand dramatize the following points:

- a. Dislocations marked by decrease the lattice friction stress.
- b. Increased twist per unit length and increased lattice curvature increase the dislocation density.
- c. Changing the sign of a dislocation, by shifting one screen from in front to in back of the other, changes the sign of the dislocation motion for the same kind of shear deformation.
- d. Increasing the dislocation density markedly decreases the dislocation velocity for a given strain rate.

These "desk top" experiments on dislocations can be obtained from Professor Frank A. McClintock, Dept. of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass. 02139.

The presence of dislocations can be readily revealed under the microscope using lithium fluoride crystals. A kit is available* entitled "Dislocations in Lithium Fluoride" by C.W. A. Newey and R.W. Davidge that gives experiments dealing with the nature of dislocations and their movement during plastic deformation and after annealing.

* Metallurgical Services, Inc.
M.P.O. Box 1075
925 Main Street
Niagara Falls, New York 14302

C. Materials Testing and Evaluation

Practically all branches of engineering are intimately concerned with materials whose properties must be determined by tests. Successful mass production depends upon inspection and control of the quality of manufactured products, which implies a system of sampling and testing. The preparation of adequate specifications and the acceptance of material purchased under specifications involve an understanding of methods of testing and of inspection. Settlement of disputes regarding failures and substandard quality almost invariable calls for investigations involving physical tests. Engineering research and development function in large measure on an experimental basis and call for carefully planned, well-devised tests.

Common purposes of materials research are to:

1. obtain new understanding of known materials,
2. discover the properties of new materials,
3. develop meaningful standards of quality or test procedures.

In addition, there may be the specific objective of choosing a material for a particular use, of determining principles to improve design with a chosen material, or of studying the behavior of the part or structure after it has been made. Although many investigations are more or less routine in nature, there are also many that call for a wide variety of tests and measurements, require an appreciation of all phases of the general problem, and make extreme demands on the skill, ingenuity, and resourcefulness of the experimenter if success is to be attained.

For the intelligent appraisal and use of the results of tests, it is important for engineers, even those not engaged in actual testing work, to have a general understanding of the common methods of testing for the properties of materials, and of what constitutes a valid test. Further, in dealing with the specification and acceptance of materials, an understanding

of the limitations imposed by methods of testing and inspection is important.

Some of the more important mechanical tests that should be included in the materials testing laboratory include:

1. Tension Test - The tensile properties of a wide variety of materials can be measured and compared. The following two references show how to get the maximum possible information from the tensile test: a. "Some Observations on the Tensile Testing of Ductile Metals," P.M. Caddell and J.H. Lamble, Bull. Mech. Engng. Educ., 8 (3), 227 (1968) and b. "Strain Hardening: An Introduction to a Fundamental Experiment," R.M. Caddell and R. Sowerby, Bull. Mech. Engng. Educ., 8, 31 (1969).

2. Compression Test - The compressive stress-strain curve of such materials as cement, brass, mild steel, cast iron, and bakelite can be determined and compared to the tensile stress-strain curve.

3. Hardness Test - The Brinell and Rockwell hardness numbers can be measured on the same materials whose tensile properties were determined. Since in a number of materials, especially commonly used carbon and alloy steels, the tensile strength in psi is approximately 510 times the Brinell hardness number, the students can test this empirical relation for their materials.

4. Bending Tests - Three or four point bending test can be conducted on glass samples. The fracture strength of glass can be measured as a function of surface condition or strain rate (see preceding section on the mechanical behavior of materials).

5. Impact Test - The Charpy or Izod impact values of mild steel as a function of heat treatment can be measured. Also, the impact values of mild steel and copper can be obtained over a range of temperatures by placing the specimens in either some cool liquid, e.g. alcohol and dry ice, or a furnace just prior to testing. The test should be completed within 5 seconds

after removing the specimens from the coolant or furnace.

6. Creep Test - The room temperature creep curves of lead and lead-tin solder can be measured with apparatus easily built in the laboratory. If a tubular furnace is available,* these experiments can be extended to include temperature and the creep of other metals such as aluminum.

7. Fatigue Test - The dependence of fatigue strength on surface finish can be measured by determining the number of cycles to failure at a given applied cyclic stress on steel specimens of various surface finishes.

8. Analysis and Presentation of Data - All laboratory programs involving materials testing should give the student an introduction to the statistical analysis of the data. This is especially important in the fatigue test and in the bend test of glass.

The following references are particularly good at discussing the principles involved in the various tests:

1. The Testing and Inspection of Engineering Materials, H.E. Davis, G.E. Troxell, and C.T. Wiskocil, McGraw-Hill Book Co. (1964).
2. Engineering Materials Science, C.W. Richards, Wadsworth Publishing Co. (1961).
3. Mechanical Metallurgy, G.E. Dieter, McGraw-Hill Book Co. (1961).
4. Engineering Materials: Their Mechanical Properties and Applications, J. Marin, Prentice-Hall, Inc. (1952).

In conclusion, the benefits to the student from a laboratory program in materials testing are training in methods of observation, knowledge of accepted methods of testing, appreciation of the significance of data derived from

* Students can build one without too much difficulty, e.g. see A.U. Seybolt and J.E. Burke, Procedures in Experimental Metallurgy, John Wiley and Sons (1953).

tests, familiarity with the properties of common materials, and an understanding of the principles involved in each test.

D. Mechanical Processing of Materials

Metal cutting is one of the more important production processes since it features either directly or indirectly in the manufacture of most of the products used in our civilization. An excellent series of experiments which illustrate most of the important phenomena occurring in the metal cutting process and which require a minimum of equipment is given in a paper "A Laboratory Course in Metal Cutting," G. Boothroyd and J.A. Bailey, Bull. Mech. Engng. Educ., 5, 3 (1966). The experiments described include:

1. study of chip formation,
2. calibration of a two component cutting force dynamometer,
3. effect of speed and feed in orthogonal machining,
4. effect of tool rake angle in orthogonal machining,
5. effect of the application of a lubricant to dry cutting conditions,
6. tool wear,
7. roughness of turned surfaces,
8. measurement of cutting temperature.

Descriptions of other production process tests in which the pertinent forces and geometry are measured are given in Manufacturing Analysis, N.H. Cook, Addison-Wesley, p. 218 (1966). These test include:

1. drilling, reaming, and tapping,
2. punching,
3. wire drawing,
4. bending.

Experiments involving the extrusion of metals are described in "Some Experiments in Metal Forming," P.J. Thompson, Bull. Mech. Engng. Educ., 5, 327 (1966).

Friction is involved in most production processes and a relative simple but accurate measurement of the friction coefficient is given in "Simple

Method for the Determination of the Coefficient of Sliding Friction,"

G. Boothroyd, Bull. Mech. Engng. Educ., 9 (3), 219 (1970).